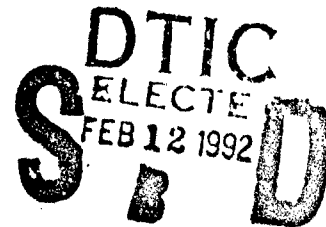AD-A245 817

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SLIP CASTING AND GREEN BODY EVALUATION OF 6% YTTRIA, 2% ALUMINA SILICON NITRIDE

JANET QUINN
CERAMICS RESEARCH BRANCH

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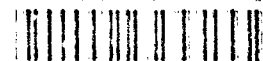||||||||||||||||||

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ABSTRACT

Silicon nitride is an excellent candidate for many engineering ceramics. Slip casting is a promising method for forming these ceramics.

This study examines some of the parameters relevant to slip casting and the green state of silicon nitride. Of these parameters, viscosity was found to be one of the most important. Generally, it is desirable to keep the slurry viscosity low with the solids content as high as possible. Outside of particle size and shape, the primary viscosity controlling factor is the chemistry of the interface between the silicon nitride particles and the suspending medium. The roles played by pH adjustments and dispersing agents in this regard are examined. Viscosities and green densities for different solids loading were determined. The ultrasonic moduli was also determined.

A relatively narrow range of slurry solids concentrations resulted in good, uncracked green bodies. A linear relationship between the ultrasonic moduli and the green density was observed.

CONTENTS

INTRODUCTION

The superior high-temperature properties of silicon nitride are well known. It has good strength, excellent high temperature strength, oxidation resistance, and is resistant to thermal shock. These properties make silicon nitride a good choice for many structural ceramics. Additionally, its high thermal conductivity and low dielectric constant make it a possible candidate for electronic substrates.

Knowledge in the areas of characterization, machining, and proper firing parameters is growing and crucial to the production of modern ceramics. Such knowledge also makes it increasingly evident that a poor initial green body cannot result in a good final product. Many fabrication problems can be traced back to the processes such as casting and green body preparation and attention is being concentrated in these areas.[1-4]

Slip casting is one of the more promising methods of producing complicated ceramic shapes and is easily accomplished in a laboratory setting. Many of the parameters associated with slip casting are relevant to other processes such as pressure casting, and to a lesser extent, injection molding and extrusion.

This study examines some of the parameters relevant to slip casting and the green state of silicon nitride. Of these parameters, viscosity was found to be one of the most important. Generally, it is desirable to keep the slurry viscosity low with as high solids loadings as possible. Outside of particle size and shape, the primary viscosity controlling factor is the chemistry of the interface between the silicon nitride particles and the suspending medium.[5] The roles played by pH adjustment and dispersing agents in this regard are examined. Viscosities for different solids loadings were determined and green body densities obtained. Characterization also includes ultrasonic nondestructive testing and mercury porosimetry.

EXPERIMENTAL PROCEDURE

The starting powder for the bulk of these experiments is UBE-SN-E-10.* Another similar powder, TOSOH-TS-10,† was also used for comparison purposes in two experiments. Both powders are synthesized by the thermal decomposition of silicon diimide and both have nearly identical sedigraph particle size distribution curves, chemical analysis, surface areas, and alpha phase content, as listed in Table 1.

The silicon nitride powder was combined with 6% yttria, 2% alumina to form a slurry with distilled water and either a commercial dispersant or ammonium hydroxide for pH control. The 6% yttria, 2% alumina slurries were mixed overnight in plastic Nalgene jars. Silicon nitride milling media were added to the containers to aid mechanical dispersion. The slurries were de-aired approximately 10 minutes in vacuum before testing and pouring.

*UBE Industries, Ltd., 7-2, Kasumigaseki 3-Chome, Chiyoda-Ku, Tokyo 100, Japan.
†TOSOH Corp., Manyo Plant 4560, Tonda, Sinnanyo-shi, Yamaguchi-ken 746, Japan.

1. TORRE, J. P., and BIGAY, Y. *Fabrication of Silicon Nitride Parts by Slip Casting*. Cer. Eng. Sci. Proc., v. 7-8, 1986 (Cocoa Beach Conf.)
2. PERSSON, M., HERMANSSON, L., and CARLSSON, R. *Some Aspects of Slip Casting Silicon Nitride and Silicon Carbide* in Ceramic Powders, Elsevier Scientific Publishing Co., Amsterdam, Holland, 1983, p. 735.
3. RABINOVICH, LEITNER, S., and GOLDENBERG, A. *Slip Casting of Silicon Nitride for Pressureless Sintering*. J. Materials Science, v. 17, 1982, p. 323.
4. OLAGNON, C., MCGARRY, D., and NAGY, E. *The Effect of Slip Casting Parameters on the Sintering and Final Properties of Si₃N₄*. Br. Ceram. Trans., J., v. 88, 1989, p. 75.
5. WHITMAN, P. K., and FEKE, D. L. *Comparison of the Surface Charge Behavior of Commercial Silicon Nitride and Silicon Carbide Powders*. J. Am. Ceram. Soc., v. 71 no. 12, 1988, p. 1086.

Table 1. COMPARISON OF UBE-SN-E-10 AND TOSOH-TS-10 SILICON NITRIDE STARTING POWDERS

Chemical Analysis (wt%)	UBE-SN-E-10	TOSOH-TS-10
N	38.0	38.0
O	2.0	1.0
C	0.2	0.05
Cl	0.01	0.030
Fe	0.01	0.010
Al	0.005	0.005
Ca	0.005	0.005
Mean Particle Size (μm)	0.6	0.6
Specific Surface Area, (M^2/g)	11.0	12.2
Alpha-Phase Content (%)	95	96

Since only 75 grams of powder comprised each specimen, a small sample adapter was utilized for the Brookfield viscometer measurements. The slips were poured onto plaster of paris molds to form either plates or bars, which were allowed to dry several days under ambient conditions. All green bodies were heated in air to 700°C for one hour to drive off moisture and burn out any additives. This heating cycle was completed in about seven hours.

The plates, about 5.5-cm square x 0.9-cm thick, were ultrasonically tested with dry coupling transducers to determine the longitudinal and transverse ultrasonic velocities. Young's moduli, the shear moduli, and Poisson's ratio were then calculated from these velocities. They were then broken into pieces for density measurements utilizing a mercury autoscan porosimeter. Pieces as large as five grams were used for density measurements, but actual porosimeter curves could only be obtained with smaller, one gram pieces. The limiting factors are the porosimeter sample cell size and the limited volume of mercury in the cell stem which could be absorbed by these very porous green bodies under pressure.

pH-CONTROLLED VISCOSITIES

Figure 1 indicates the change in pH of a silicon nitride slip with time. This slurry, consisting of 75 g of UBE powder and 112.5 g distilled water (40% solids), was continuously mixed in a plastic jar containing silicon nitride milling media. No additives were present in this powder-water mixture. The pH was periodically obtained during the course of four days and was found to rise most rapidly during the first few hours.

This pH increase with time, accompanied by a corresponding decrease in viscosity, probably contributes to the popularity of "aging" slips in their water environment before casting.
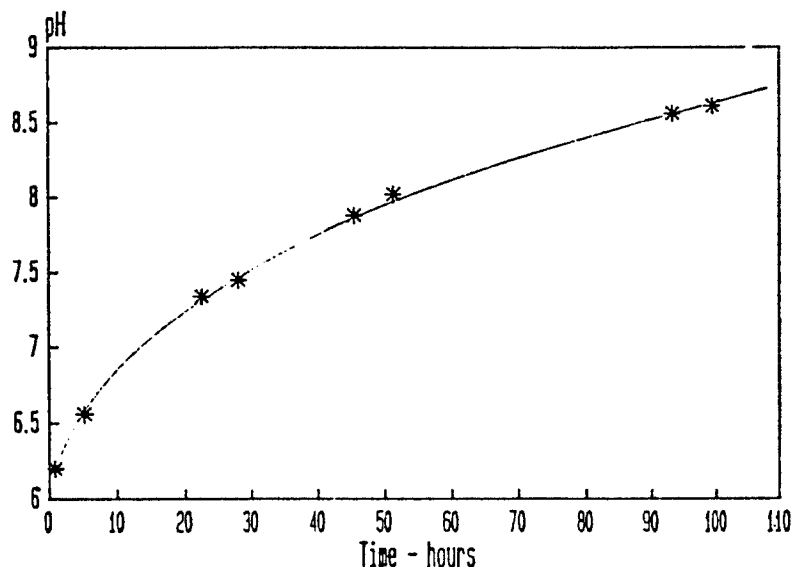
Figure 1. pH versus mixing time 'or a 40% UBE silicon nitride - 60% distilled water slurry.

This rise in pH with time was also noted by Pivinskii, et al.[6] who suggest the nitride decomposes in an initial time period of three to five hours, after which there is no additional rise in pH.

The Figure 1 data confirms the pH increase, but at a much slower rate. Indeed, a pH measurement taken of the Figure 1 slurry three days after the final test indicated that after 215 hours the pH was still rising slightly (pH = 9.49 at 215.22 hours).

P. Greil et al.[7] refer to the aging effect and explain the pH change in terms of surface composition and impurity content of the silicon nitride starting powders. They relate an increase in pH to a high dissociation constant for the nitride decomposition proposed in Reference 6, but note that a silica-like surface area would cause a pH drop due to the hydrolysis of silica and disassociation of silicon acid.

The pH change of a silicon nitride slip thus represents a balance between the hydrolysis of the pure silicon nitride and the hydrolysis of the silica surface layer.

Powder surface treatments such as preoxidation and chemical leaching would increase the availability of silicon-oxygen compounds on the particle surfaces and lower the pH. Such processes result in improved slips,[8] but sintering parameters and/or the final product may be affected. Obviously, manufacturers would prefer not to pretreat but to use the starting powders as-received. Careful characterization then becomes necessary, for the as-received particle surface chemistry will dictate processing parameters and achievable viscosities.

6. PIVINSKII, YU. E., PODOBEDA, L. G., BURAVOV, A. D., and POLUBATONOVA, L. P. *Some Properties of Aqueous Silicon Nitride Suspensions*. Poroshkovaya Metallurgiya, no. 3 (159), March 1976, p. 37.

7. GREIL, P., NAGEL, A., STADELMANN, H., and PETZOW, G. *Review; Colloidal Processing of Silicon Nitride Ceramics*. Ceramic Materials and Components for Engines, p. 319.

8. STADELMANN, H., PETZOW, G., and GREIL, P. *Effects of Surface Purification on the Properties of Aqueous Silicon Nitride Suspensions*. J. European Ceramic Society, v. 5, 1989, p. 155.

The high pHs attained by the Figure 1 slurry would indicate the presence of very little silica, or rather a relatively thin silica-like surface layer on the UBE starting powder. The continuing rise of pH with time might correspond to the exposure of new silicon nitride surface areas, either from the breakup of the silica-like particle coating or the breakup of the particles themselves. The latter is likely to be negligible in a mixing rather than a milling operation.

An indication of the amount of silicon oxygen compounds on the particle surfaces of the UBE powder of this study was determined by an oxygen analysis performed by Luvik, Inc. of Boylston, MA. A slurry was prepared identical to the slurry of Figure 1. Eight samples were withdrawn from this second slurry over the course of several days. These samples were dried and the resulting powder analyzed for oxygen content. Test results indicated no significant change in oxygen with either pH or time during the course of this experiment. All samples indicated about 1.37% oxygen, consistent with the 1.3% oxygen listed in the UBE data sheets. Of this amount of oxygen present in the samples, approximately 70% is estimated to be concentrated at the particle surfaces in powders processed by the UBE route.[9] The powders of this study, therefore, have a relatively thin silica-like particle coating accounting for approximately 0.96% oxygen impurity.

The graph in Figure 2 demonstrates the dependence of slip viscosities on pH for the as-received UBE powder with the 6% yttria, 2% alumina sintering aids. It is well known that slip viscosities will drop with either very high or very low pH, as powder particles of either positive or negative charge will repel each other. Zeta potential measurements as a function of pH can be used to determine optimum slurry conditions. Highly negative and highly positive zeta potentials correspond to highly charged particles and good dispersion, with the worst slurry conditions occurring at a zeta potential near zero. For silicon nitride, the zeta potential isoelectric point occurs at a pH of 5 to 6 (see Reference 7, 8, and 10). High zeta potentials correspond to pHs below 3 and above 9. Slips are not generally produced in the low pH range since the viscosities are optimized at such highly acidic slips that equipment and mold lifetimes are significantly reduced. Other considerations include the effectiveness of commercial processing additives and compatibility of sintering aids in an acid environment.

The Figure 2 basic pHs were adjusted with ammonium hydroxide. As the solids loadings increase, the Figure 2 curves become steeper and move to the right. Higher pHs are necessary to obtain optimal low viscosities as the particles pack closer together. At the highest solids concentrations, small changes in pH result in large viscosity changes until minimum viscosities are achieved.

Silicon nitride slips are notoriously pseudoplastic (see Reference 2, 4, and 11). The extent of this shear-thinning is evident in Figure 3, where higher shear rates result in lower measured slip viscosities. Since shear rate sensitivity decreases at higher values, there is an advantage to working at higher shear rates for data comparison and reproducibility. Also, thixotropic effects are reduced at higher shear rates.[11] Subsequent figures in this study include data at different shear rates to further characterize the shear thinning effect.

9. DANFORTH, S. C., and HAGGERTY, J. S. J. Am. Ceram. Soc., v. 66, 1983, c. 58.

10. SHAW, T. M., and PETHICA, B. A. Preparation and Sintering of Homogeneous Silicon Nitride Green Compacts. J. Am. Ceram. Soc., v. 69, no. 2, 1986, p. 88.

11. MOORE, F. The Rheology of Ceramic Slips and Bodies. Br. Ceramic Research Association 10-28-58 (try Trans. Brit. Ceram. Soc., starting '59).
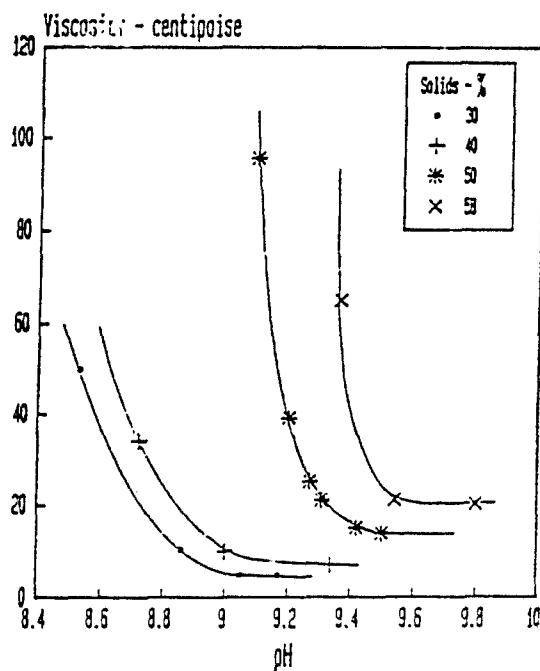
Viscosity - centipoise

Figure 2. Viscosity versus pH for silicon nitride slurries at shear rate = 39.6/sec. As the solids concentrations increase, the curves become steeper and higher pHs are necessary to obtain optimal, low viscosities. The lowest viscosities for each solids concentration is plotted in Figure 4.
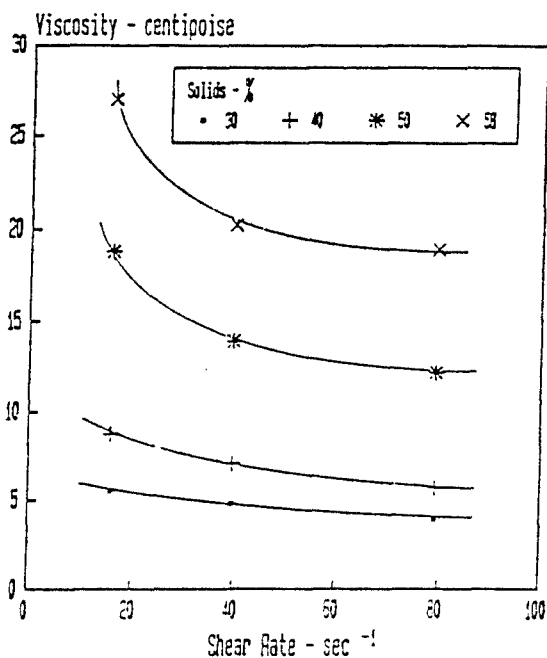
Viscosity - centipoise

Figure 3. Viscosity versus shear rate. The viscosities are less shear-sensitive at higher shear rates and lower solids concentrations.

5

Figure 4 indicates the lowest attained viscosities for the UBE silicon nitride with 6% yttria, 2% alumina slurries. These viscosities were obtained by noting the pH and solids concentrations of the minimum viscosities in Figure 2. The left axis indicates the minimum pHs needed to attain the viscosities while the right axis indicates the corresponding solids content. The pH values rise at approximately the same rate as the solids concentrations within the range exhibited in Figure 4. As can be seen, the curve starts to flatten at about 60% solids concentrations. Additional solids result in large viscosity increases, limiting slip casting to solids concentrations under 65% for this type of slurry.
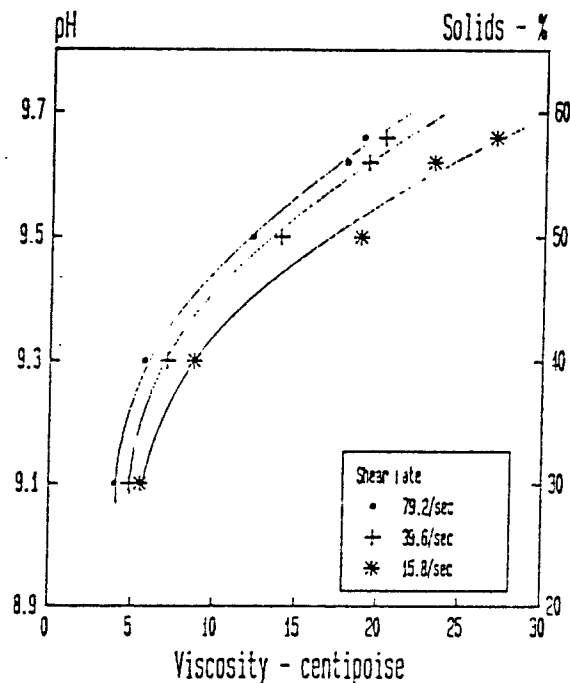
Figure 4. Lowest attained viscosities for solids concentrations (right axis) at the corresponding pHs (left axis). The middle curve (shear rate = 39.6/sec) can be determined from the minimum viscosities in Figure 2.

SLIPS WITH COMMERCIAL ADDITIVES

A commercially available dispersant, Darvan C,* was added to several slips. Darvan C is an ammonium polymethacrylate aqueous solution containing 25% solids. Varying amounts of this liquid, about 0.2% to 0.4% of the slurry solids content, was found to optimize the slip viscosities. The slips containing the Darvan C additive behaved similarly to slips without the additive but at lower pHs. Shear-thinning behavior was still evident. As can be seen in Figure 5, the effect of this additive was to lower the pH at which the minimum viscosity was reached.

An advantage to using Darvan C is easier viscosity control since a small amount of this additive lessens the need for the pH - controlling ammonium hydroxide which is volatile (and pungent). Also, the more highly basic slips can be detrimental to equipment and molds.

*R. T. Vanderbuilt Co., Norwalk, CT.

Finally, the zeta potentials of the yttria and alumina sintering aids are closer to the silicon nitride zeta potential in the less basic Darvan C slips. A disadvantage is the inclusion of a burn-out step in the removal of this additive, although residual ash (0.04 wt%) is minimal.[12]
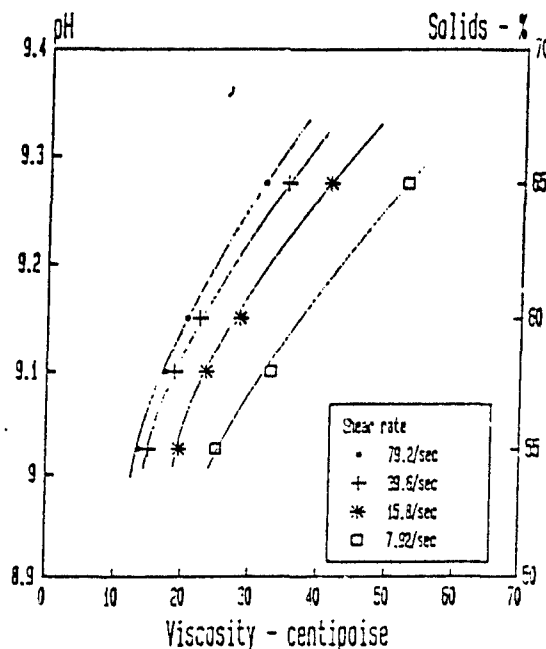

Figure 5. Lowest attained viscosities for Darvan C slurries for solids concentrations (right axis) at corresponding pHs (left axis). Comparison with Figure 4 indicates the same optimal viscosities are achieved at lower pH values.

Approximately 0.05% of another commercial dispersant, Lignosol NST-150,* was added to some slips. Slurries containing this lignosulphonate wood derivative did not exhibit uniform rheological behavior. Some of the slips with high solids concentrations were rheopectic, but this could be exacerbated by the small sample sizes which dry quickly. Shear-thinning was much reduced, and a slurry containing the very low solids concentration of 35% was dilatant at the lower shear rates. This is illustrated in Figure 6. The Figure 6 axes are the same as the Figure 3 axes for easier comparison. Reference 2 also notes a marked shear-sensitivity decrease in slurries containing a lignosulphonate dispersant.

Any advantage to using lignosulphonate dispersants must be weighed against the difficulty of burning the additives out of the resultant green bodies. A crucible containing Lignosol NST-150 was heated to 800°C for three hours in air after which 0.71% solid residue yet remained.

Four slips were prepared with 0.6% Polyglycol E400,† a plasticizer. Green bodies containing this additive seemed somewhat less prone to cracking, but the liquid slips were very slightly more viscous. This can be seen in Figure 7, which compares the resulting viscosities of all the slurries of this study.
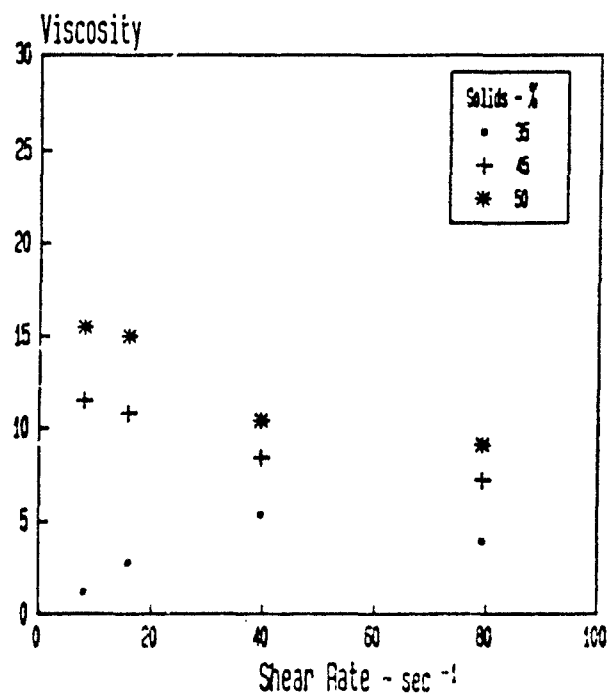
Figure 6. Viscosity versus shear rate for a slurry containing Lignosol NST 150. The anomalous rheological behavior is apparent in comparison with Figure 3.
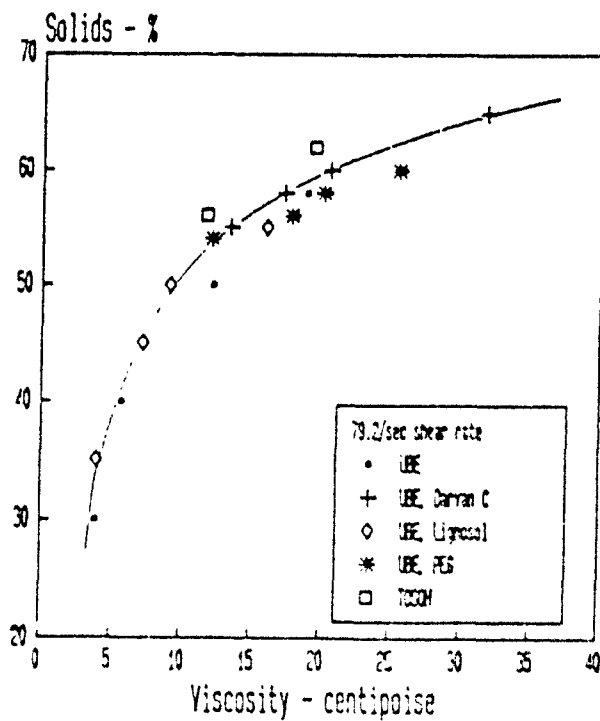

Figure 7. Solids concentration versus viscosity. The better slurries (high solids, low viscosities) were obtained with the TOSOH starting powder, whereas the slurries with the polyglycol additive (PEG) fell in the poorer region beneath the curve.

8

Two slips were prepared from an as-received TOSOH silicon nitride powder in an identical fashion as the UBE slips of Figures 3 and 4. The pH-controlled viscosity data from these two slips are also included in Figure 7. All the Figure 7 slurries contain the 6% yttria, 2% alumina sintering aids. The single, high shear rate of 79.2/sec was chosen to more easily compare the data.

All the slurries tend to follow a general curve, with the polyglycol additive (PEG) slurries slightly under the curve, and the TOSOH slurries slightly above the curve in a higher, more desirable region. Whether controlled by pH alone (ammonium hydroxide), or with the aid of dispersants, the slurries of the as-received starting powders all have limiting viscosity and solids concentration values within 3% of the Figure 7 curve. Changes in powder surface treatments or particle shape, size, or distribution appear to be necessary to significantly alter this curve.

GREEN BODY CHARACTERIZATION

Slip casting requires a low viscosity for the slurry to be poured and dewatered, especially for intricate shapes. At the same time, a high solids concentration is necessary to minimize shrinkage and achieve a dense, sinterable green body.

Cracking was evident in green bodies cast with solids concentrations under 52% and exceeding 62% with or without the commercial dispersants. It was noted that the cracking was somewhat abated in slips containing the PEG plasticizer and in slips which were dried in a high humidity, low temperature environment.

Densities and porosimeter curves were obtained for green bodies cast of slips within the 54% to 62% solids range and dried under ambient conditions. Two of the slips were comprised of the TOSOH starting powders, the rest of the UBE powders. Four of the UBE slips contained about 0.3% Darvan C, and four other UBE slips contained 0.6% Polyglycol E400.

Densities of small, porous green bodies are not easily measured. Geometric densities are not adequate for very small samples, especially if the shapes are not exactly regular. Combinations of coatings and the water-Archimedes method proved difficult to implement, although Reference 13 describes a glucose-Archimedes method which is successful with green bodies of low porosity.[13]

Densities were obtained by placing each specimen in a porosimeter sample cell. Mercury was introduced into the cells under atmospheric pressure. The measured sample weights and the combined mercury-sample weights, together with the known cell volumes and mercury density, were utilized to calculate the sample densities. These densities were plotted against solids concentrations. The somewhat scattered results comprise Figure 8 from which a few general trends can be noted.

The green body densities in this study generally rise with solids concentrations, which is to be expected. The better densities were achieved with the TOSOH starting powders, and the worst with the slurries containing the PEG additive. This detrimental effect might be offset, however, if cracking could be controlled by this additive such that a higher solids loading were possible.

13. PENNING, F. C. M., and GRELLNER, W. *Precise Nondestructive Determination of the Density of Porous Ceramics.* J. Am. Ceram. Soc., v. 72, no. 7, 1989, p. 1268.

Solids - %

Legend:
- · UBE
- + UBE + Darvan C
- * UBE + PEG
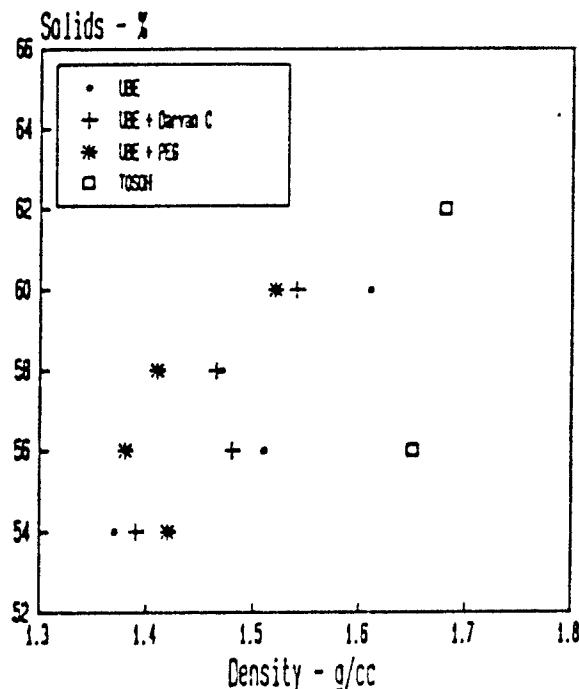- □ TOSOH

Density - g/cc

Figure 8. Green body density versus solids concentration. The somewhat scattered results indicates a trend for green densities to increase with solids concentrations.

The green body densities were confirmed in an interesting way. The intruded volumes of mercury were obtained from the porosimeter curves of all the green bodies. The intruded volume was added to the volume occupied by the green body solids, assuming the solids density to be 3.28 g/cc. (This composite density is obtained for a 6% yttria, 2% alumina, 92% silicon nitride with individual densities of 5.03, 3.99, and 3.20 g/cc, respectively.) The sample weight was then divided by the combined volumes. The resulting values comprise Table 2 where they are compared to the densities previously obtained by the modified mercury Archimedes method. It is readily apparent that the densities calculated from mercury intrusion agreed very well with the more conventionally obtained densities. In many cases, the values match to all three significant digits. The minor discrepancies occur at the higher densities for several reasons. One is that the sample passageways become so small that higher applied pressures must be utilized for more complete mercury intrusion. The porosimeter curves flatten so gradually at these higher pressures, it is difficult to estimate the final total intrusion values. It is even possible that the higher density green bodies contain a very small volume of porosity which is not surface connected. These possibilities favor an error which would give artificially high density values, which is the case here. Nevertheless, the close agreement of the densities in Table 2 indicates the Figure 8 scatter is more likely due to inconsistencies in pH and powder measurements than green density measurements.

Figures 9a and 9b are representative porosimeter curves for samples of the same mass but different densities. The Figure 9a graph illustrates the trend for mercury to enter the low density samples under a low pressure, followed by a steep intrusion curve which levels off quickly. The higher density curves, however, start at higher initial pressures, then rise and level off more gradually, as illustrated in Figure 9b.

Table 2. COMPARISON OF DENSITIES CALCULATED BY TOTAL INTRUDED MERCURY (SAMPLE MASS/COMBINED VOLUME) AND BY THE MODIFIED MERCURY-ARCHIMEDES METHOD (CONVENTIONALLY MEASURED DENSITY)

Sample Description	Porosimeter Sample Mass (g)	Intruded Volume (cc)	Solids Volume (cc)	Sample Mass Combined Volume (g/cc)	Conventionally Measured Density (g/cc)
UBE	0.911	0.3795	0.2779	1.37	1.37
UBE	0.864	0.3195	0.2633	1.48	1.47
UBE	1.265	0.4526	0.3857	1.51	1.51
UBE	1.296	0.4020	0.3951	1.63	1.61
UBE, Darvan C	0.923	0.3823	0.2814	1.39	1.39
UBE, Darvan C	1.083	0.4048	0.3302	1.47	1.47
UBE, Darvan C	1.070	0.3921	0.3262	1.49	1.48
UBE, Darvan C	1.253	0.4245	0.3820	1.55	1.54
UBE, PEG	1.02	0.4525	0.3110	1.34	1.38
UBE, PEG	1.13	0.4554	0.3445	1.41	1.41
UBE, PEG	1.48	0.5692	0.4512	1.45	1.42
UBE, PEG	1.30	0.4610	0.3964	1.52	1.52
TOSOH	1.365	0.4252	0.4161	1.62	1.65
TOSOH	1.033	0.292	0.3149	1.70	1.68
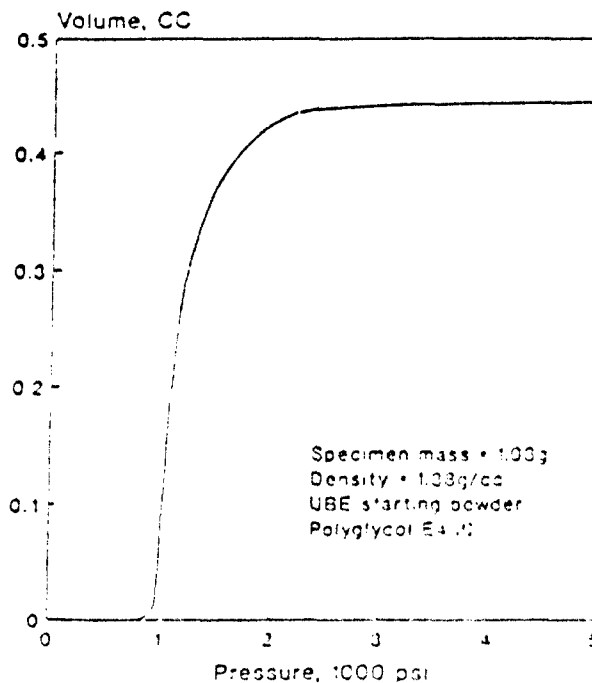
Figure 9a. Intruded Hg volume versus pressure for a low density sample. The low pressure at which intrusion begins (c1000 psi) followed by rapid complete intrusion is typical of the low density green bodies of this study.

Volume, CC

Specimen mass = 1.03g
Density = 1.68g/cc
TOSOH starting powder
No Additives
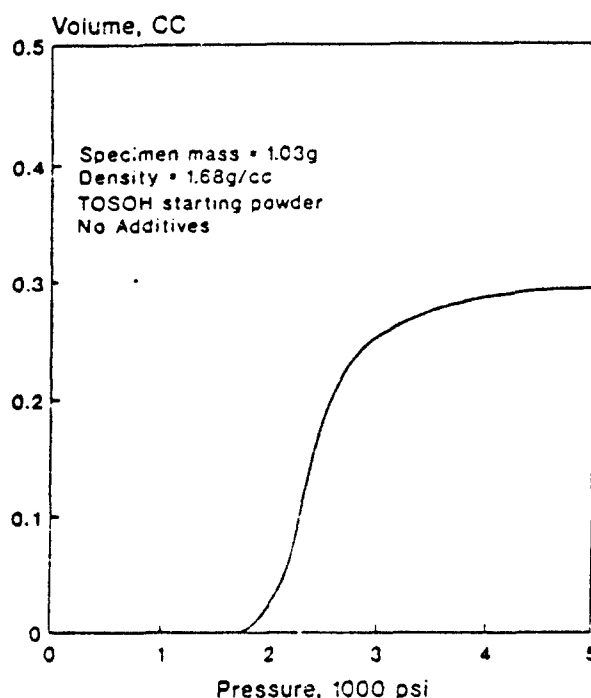
Pressure, 1000 psi

Figure 9b. Intruded Hg volume versus pressure for a higher density sample.
Mercury intrusion begins at a higher pressure (c2000 psi) and levels off more
slowly, indicating the narrower and more distributed particle spaces typical of
the higher density green bodies of this study.

From this data it can be inferred that the green bodies have no channels or passageways larger than the size associated with the initial intrusion pressure. This does not mean that very large pores do not exist within the green bodies; it is only the size of the interconnecting passageways which affect the intrusion pressures.[14] Denser green bodies have narrower median passageways, but a much wider passageway size distribution, as inferred by the flatter, more elongated porosimeter curves.

These observations are illustrated in Figure 10. The lower curve indicates the pressures at which the mercury first intrudes the samples. The upper curve is the median pressure at which the greatest influx of mercury occurs.

It is apparent in Figure 10 that the intrusion pressures rise rapidly as the green densities approach 1.7 g/cc. Perhaps specimen cracking beyond these densities are due to difficulty in dewatering the slurries as the interparticle passageways become too narrow or even nonexistent.

Ultrasonically determined moduli were obtained for the green bodies of Figure 10. Young's modulus, E, and the shear modulus, G, were calculated from the following equations:[15]

14. LEE, H. H. D. *Validity of Using Mercury Porosimetry to Characterize the Pore Structures of Ceramic Green Compacts.* J. Amer. Ceram. Soc., v. 73, no. 8, 1990, p. 2305.
15. MCGONNAGLE, W. J. *Nondestructive Testing.* Gordon and Breach, Science Publishers, Inc., New York, NY, 1961, p. 211.

$$E = \rho V_{\ell} (1+\nu) (1-2\nu)/(1-\nu) \tag{1}$$

$$G = \rho V_t$$

where ρ = material density, V_{ℓ} = longitudinal wave velocity, V_t = transverse or shear wave velocity, and ρ = Poisson's ratio = $(E/2G) - 1$.
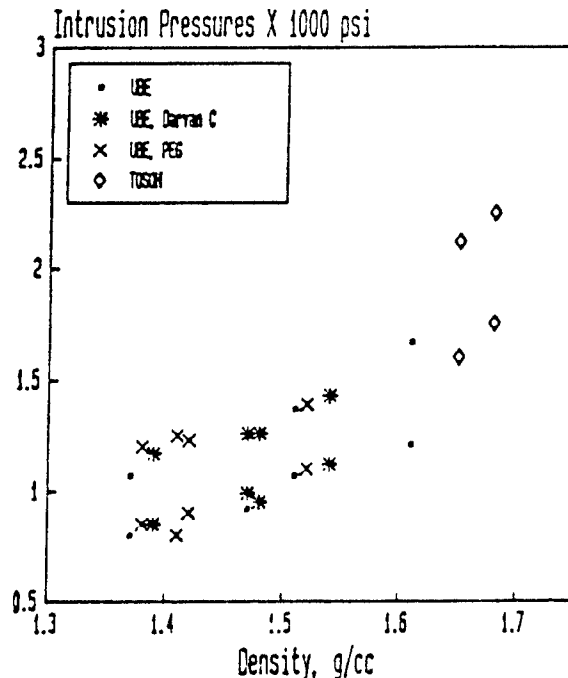
Figure 10. Intrusion pressures versus density. The initial and median intrusion pressures rise more quickly with density as the channels between particles become narrower.

These equations are commonly employed with fully dense, isotropic materials, but evidence of validity with porous sintered ceramics has recently been established.[16] It has not yet been fully determined that the moduli in ceramic green bodies can be accurately calculated by the previous formulae. Nevertheless, linear relationships between the acoustic moduli and green body densities in alumina and steatite have been demonstrated.[17] Linear relationships were also found for the silicon nitride green bodies of this study, as illustrated in Figure 11. From this figure it can be seen that most of the combined density data fall on single straight lines, with the exception of the specimens which contained the Polyglycol E400 additive. These specimens fall on separate, lower lines. Evidently, ultrasonic velocities are sensitive to differences in green bodies processed with this additive. The differences are likely structural and could affect subsequent sintering. A more detailed analysis of the ultrasonic data is presented in Reference 17.

16. BHARDWAJ, M. C. *Simple Ultrasonic NDC for Advanced Ceramics Development and Manufacture*. Proceedings of International Process Modeling and Mechanical Behavior, Anaheim, CA, February 19-22, 1990.

17. QUINN, J., and TARDIFF, L. *Ultrasonic Evaluation of Silicon Nitride Green Bodies*. U.S. Army Materials Technology Laboratory, MTL TR 91-19, May 1991.
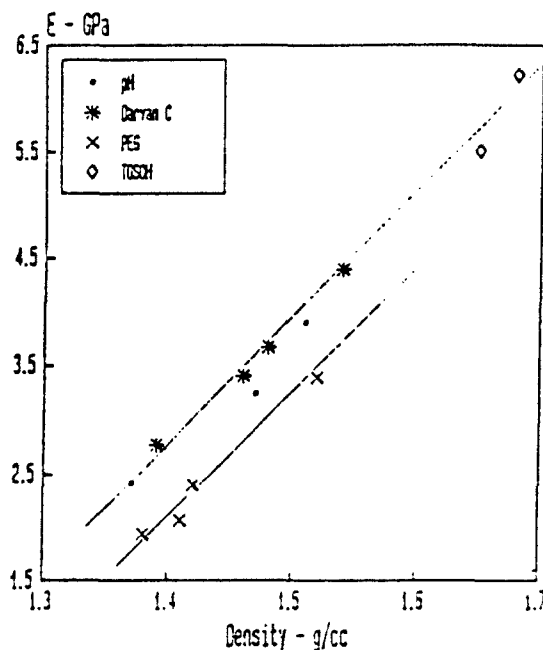
Figure 11. Ultrasonically determined Young's modulus, E, versus density.
The samples which contained the polyglycol additive (PEG) comprise the
lower line, while the other samples follow the higher line.

CONCLUSIONS

Slip casting of silicon nitride with 6% yttria, 2% alumina is controlled by numerous inter-dependent variables, including solids concentration, pH, viscosity, and commercial processing addi-tives. These variables can be optimized for starting powders in the as-received state, but initial powder characterization will dictate achievable parameters. Further optimization would necessi-tate powder surface treatments or changes in physical powder characteristics; e.g., milling.

A relatively narrow range of slurry solids concentrations resulted in good, uncracked green bodies with the upper limit of this range producing the densest samples. Porosimetry results suggest difficulty in extending this range if cracking is due to closed or narrow interparticle channels within the specimen which would hinder dewatering and drying.

Nondestructive ultrasonic testing revealed a linear relationship between ultrasonic moduli and specimen densities, and also indicated differences in specimens processed with a commer-cial plasticizer.

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SLIP CASTING AND GREEN BODY EVALUATION
OF 6% YTTRIA, 2 % ALUMINA SILICON NITRIDE -
Janet Quinn

Technical Report MTL TR 91-52, December 1991, 17 pp-
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Key Words

Silicon nitride
Processing
Characterization

Silicon nitride is an excellent candidate for many engineering ceramics. Slip casting is a promising method for forming these ceramics. This study examines some of the parameters relevant to slip casting and the green state of silicon nitride. Of these parameters, viscosity was found to be one of the most important. Generally, it is desirable to keep the slurry viscosity low with the solids content as high as possible. Outside of particle size and shape, the primary viscosity controlling factor is the chemistry of the interface between the silicon nitride particles and the suspending medium. The roles played by pH adjustments and dispersing agents in this regard are examined. Viscosities and green densities for different solids loading were determined. The ultrasonic moduli was also determined. Viscosities and green densities for different solids loading were determined. The ultrasonic moduli was also determined. A relatively narrow range of slurry solids concentrations resulted in good, uncracked green bodies. A linear relationship between the ultrasonic moduli and the green density was observed.